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EXPERIMENT (5)

ASTM Distillation

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Distillation is the method of separating the components of a solution which depends upon the distribution of the substances between a gas and a liquid phase .Distillation may be done in two ways :

1. Production of vapor by boiling the liquid mixture to be separated and condensing the vapor without allowing any to return to the still ,there is no reflux.

2. Returning a part of condensate to the still under such conditions that this returning liquid is brought into intimate contact with on their way to condenser.

The distillation column consists of several trays, which allow the simultaneous travel of liquid down the tray and vapor up the tray, allowing mixing of the two phases and therefore equilibrium

The liquid mixture that is to be processed is known as the feed. The feed-tray divides the column into a top (enriching or rectification) section and a bottom (stripping) section.

The vapor moves up the column ,and as it exits the top of the unit, it is cooled by a condenser. The condensed liquid is stored in a holding vessel known as the reflux drum. Some of this liquid is recycled back to the top of the column and this is called the reflux.

The condensed liquid that is removed from the system is known as the top product or distillate

The basic test method of determining the boiling range of a petroleum product by performing a simple batch distillation has been in use as long as the petroleum industry has existed. It is one of the oldest test methods under the jurisdiction of ASTM Committee D02, dating from the time when it was still referred to as the Engler distillation. Since the test method has been in use for such an extended period, a tremendous number of historical data bases exist for estimating end-use sensitivity on products and processes. The distillation (volatility) characteristics of hydrocarbons have an important effect on their safety and performance, especially in the case of fuels and solvents. The boiling range gives information on the composition, the properties, and the behavior of the fuel during storage and use.

Volatility is the major determinant of the tendency of a hydrocarbon mixture to produce potentially explosive vapors. The distillation characteristics are critically important for both automotive and aviation gasoline's, affecting starting, warm-up, and tendency to vapor lock at high operating temperature or at high altitude, or both. The presence of high boiling point components in these and other fuels can significantly affect the degree of formation of solid combustion deposits. Distillation limits are often included in petroleum product specifications, in commercial contract agreements, process refinery/control applications, and for compliance to regulatory rules. This test method can applied to contaminated products or hydrocarbon mixture. This is valuable for fast product quality screening , refining process monitoring , fuel adulteration control, or other purpose including use as a portable apparatus for field testing .This test method can uses an automatic micro distillation apparatus privies fast result using small sample volume , and eliminates much of the operator time subjectivity in comparison to test method D86 .

This test methods covers a procedure for determination of the distillation of characteristics of petroleum products and liquid fuels having boiling range between 200c to 4000c at atmospheric pressure using and automatic micro distillation apparatus .The test method also applicable to hydrocarbons with a narrow boiling range , like organic Solvent or oxygenated compounds. The method designed for analysis of distillate products; it is not applicable to Product appreciable quantize of residual material.

Some examples of ASTM (American society for testing and material) standards utilizing distillations are; D86 Test method for distillation of petroleum products at atmospheric pressure, D20-03 Standard test method for distillation of road tars, D1106 Test method for distillation of petroleum products at reduced pressure, D 323 Test Method for Vapor Pressure of Petroleum, D 396 Specification for Fuel Oils, D 850 Test-Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials, D 975 Specification for Diesel Fuel Oils, D 1078 Test Method for Distillation Range of Volatile Organic Liquids, D 2892 Test Method for Distillation of Crude Petroleum, D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products, D 4 177 Practice for Automatic Sampling of Petroleum and Petroleum Products, D 4953 Test Method for Vapor Pressure of Gasoline and Gasoline Oxygenate Blends.

The aim of this experiment is determination of boiling range characteristics of petroleum product by using ASTM distillation. ASTM Distillation is a standard Test Method for Distillation of Petroleum Products at Atmospheric Pressure.

Working Principle :

It is based on the principle of differential or simple distillation. A batch of liquid is charged to a still fitted with some sort of heating device. The charge is boiled slowly and the vapors are withdrawn as rapidly as they form to a condenser where they are liquefied and the condensate is collected in the receiver.

EXPERIMENTAL PROCEDURE :

1. The distillation flask is filled with 100 ml of sample.
2. Water in the cooling bath is replaced and cleaned properly for effective cooling. Ice is placed in the cooling bath for removal of heat produced.
3. The distillation flash and the receiving cylinder are fitted to the input and output of the cooling bath by connecting pipe. Thermometer is inserted to distillation flask.
4. To reduce evaporation loss due to leakage, the various joints in the arrangement (such as distillation flask - connection pipe and cooling bath output - receiving cylinder) are sealed properly using some paper.
5. After all arrangements are completed, heater is switched on and heat is supplied at constant and standard rate for proper vaporization.
6. The temperature is noted when first drop of liquid condensed is received at the receiving cylinder. After that temperature for each 5 ml rise in volume of distillate is noted.

7. Final boiling point temperature is noted when no further liquid is received at the cylinder, though heat is supplied at the same rate. Then we stop heating and final volume is recorded.

8. The residue is cooled, and measured using a measuring cylinder. Thus subtracting from total volume to volume distillate we get the loss.

Apparatus Used :

1. Distillation Flask
2. Cooling Bath
3. Heater
4. Thermometer
5. Receiving cylinder to collect distillate.

Fig 1 : Experimental setup

Experiment calc.

The following data are obtained by performing the experiment initial point of the product=54°C

Volume percent distilled	Temperature °C
10	65
15	69
20	74
25	78.5
30	83.5
35	90
40	94.5
45	100
50	100.4
55	109
60	114
65	119.5
70	125.5
75	133
80	141.5
85	155
90	173
92	150

Final boiling point of the product=173°C

Volume distilled =92 ml

Residue left =2ml

Evaporated =6ml

Then we draw graph ,Volume distilled in ml (X axis) & Temperature °C (Y axis)